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GRANULATION OF ORGANIC DYES FOR CASTABLE SMOKE COMPOSITIONS.(U)

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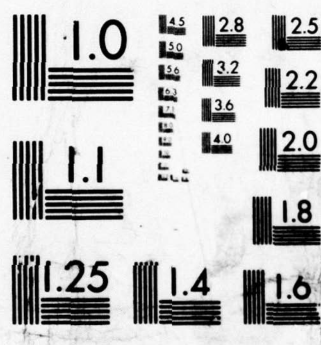


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# GRANULATION OF ORGANIC DYES FOR CASTABLE SMOKE COMPOSITIONS

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(6) GRANULATION OF ORGANIC DYES FOR CASTABLE

SMOKE COMPOSITIONS

by


(10) G. Couture ~~and~~ A. Roy


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## RESUME

La préparation de compositions fumigènes coulables, ayant une viscosité de fin de mélange de moins de 5 kilopoises (kP) pour des concentrations en solides de plus de 80% en poids, ne peut se faire que si on utilise des colorants granulés. Comme les teintures organiques entrant dans ces compositions ne se trouvent sur le marché que sous forme de poudres pulvérulentes, on a mis au point deux procédés de granulation. Le premier, par voie de fusion/cristallisation/broyage, est efficace pour les colorants ne contenant qu'un seul composé, comme le colorant orange qui a un point de fusion unique. Par contre le second procédé, par compactage/broyage, a donné de bons résultats avec tous les colorants étudiés; on peut l'utiliser en continu et produire économiquement du granulé de la grosseur désirée. On met également en lumière les propriétés du granulé et ses avantages au point de vue de la mise en oeuvre des mélanges fumigènes. (NC)

## ABSTRACT

Processing of castable smoke compositions having an end-of-mix viscosity less than 5 kilopoises and a solids loading greater than 80% by weight is only possible when granulated dyes are used. Since the organic dyes used in these compositions are commercially available only as very fine powders, two methods of granulation have been developed. The first method, by fusion, crystallization and grinding, is useful for single component dyes, such as orange, where there is a single melting point. The second method, by compaction and grinding, has, on the other hand, given good results with all the dyes tried and can be used in a continuous process to economically produce granulated material of the desired size. Also discussed are the properties of the material and its advantages with respect to the manufacture of smoke compositions. (U)

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## 1.0 INTRODUCTION

The work was performed at DREV between October 1972 and September 1974 under PCN 21B03 (formerly 05J03) Castable Pyrotechnics. Two methods were perfected to change dyes available as very fire powders into granulated material of the desired mean size.

The preparation of castable-type smoke compositions was made possible by substituting the commercial dye with granulated material having an average size of 650 μm by weight. Due to their characteristics end-of-mix viscosity, these compositions may be cast into the tubes by gravity alone. The composite smoke compositions dealt with herein consist primarily of an organic dye (Appendix), potassium chlorate ($KClO_3$) and lactose. These solids are scattered within an hydroxyl-terminated polybutadiene binder vulcanized with an isocyanate (1).

It was evident from the outset that the viscosities required for a castable product (less than 5 kP, $5 \times 10^2 N.s/m^2$) could not be obtained with the available powders, since their mean particle size was always less than 2 μm. The various surface agents used to improve mix fluidity could not lower end-of-mix viscosity below 10 kP for compositions containing 25% liquids by weight.

The viscosity of a liquid/solid suspension is closely related to the particle size distribution of the solids it contains. An appropriate mix of fine particles and of larger particles is required in order to obtain a minimum viscosity. By reducing the total surface of the solid phase with large granules and by filling the gaps between them with fine granules, optimal compaction is achieved, thus reducing the amount of liquid required to fluidify such a mix. Optimum compaction for a 2-component mix (fine and large particles) is usually obtained with approximately 65% by volume large particles and 35% fine ones. Secondly, the D/d ratio (large particle mean diameter over small particle mean diameter) has a direct effect on compaction; the larger D/d, the better the compaction (2). Since the amount of dye in smoke compositions usually ranges from 60 to 70% of the solid phase volume, any substitution of powder dye by granules will result in a considerable reduction of end-of-mix viscosity (3). An optimum solids mix will thus be obtained, with a fine particle volume of 30 to 40%. These fine particles comprise the $KClO_3$, the lactose and the other fuel ingredients except for the dye.

The following sections describe two commercial powder granulation methods and the improvement of the manufacturing properties.

## 2.0 GRANULATION BY FUSION, CRYSTALLIZATION AND GRINDING

The first method was designed for the granulation of 1-amino-anthraquinone (1-AAQ) and consists of melting, recrystallizing and grinding the commercial product. The fraction required is recovered by sieving and the remnants are remelted.

### 2.1 Description of the method

The orange dye (1-AAQ) is a relatively pure product, with a melting point of 252°C. It can thus be melted slowly without causing too much sublimation. However, occasional stirring is required in order to ensure permanent contact between the solid phase and the melting phase. Without such stirring, a cavity appears above the liquid, causing overheating of the already melted fraction and sublimation of a portion thereof. Two-litre stainless steel beakers are used, into which 1 kg of dye may be poured in successive steps. A hot plate is sufficient to melt the dye within a reasonable amount of time, while leaving the cover cool enough to recover the small fraction of dye that is sublimated. The molten dye is then poured into dismountable moulds where recrystallization at ambient temperature occurs. The 15-cm cubes thus obtained are subsequently ground to the desired size. Because of the fragility of the material, grinding must be gradual in order to prevent excessive friction that could cause an increase in the amount of fine particles.

The first operation consists of breaking the blocks into 3 to 4 cm pieces that can be fed into an ice-breaker-type blade crusher, where they are reduced to less than 1 cm. The dye is then processed through 4 pairs of 12-cm-diameter differential rollers (20 and 40 r/min). Their spacing diminishes gradually (5, 3, 1.5 and 0.75 mm) so that the gap of each roller pair is half the size of the pieces admitted. Any attempt to reduce the number of passes leads to a rapid rise in the fine particle ratio. Figure 1 illustrates that increase: if the number of passes is reduced from 4 to 3 with 5, 2 and 0.75-mm roller gaps, the amount of material,100 μm and less, increases from 20 to 30% by weight.

### 2.2 Discussion of the method

Acceptable grain size is obtained after removing the less-than-100-μm fraction by sieving. This fraction amounts to approximately 20% of the total dye quantity processed and is subsequently recirculated with the commercial dye. Figure 2 shows the dye grain size before and after sieving. Average size after sieving ranged from 300 to 500 μm, but higher figures could have been obtained by increasing the roller gap.

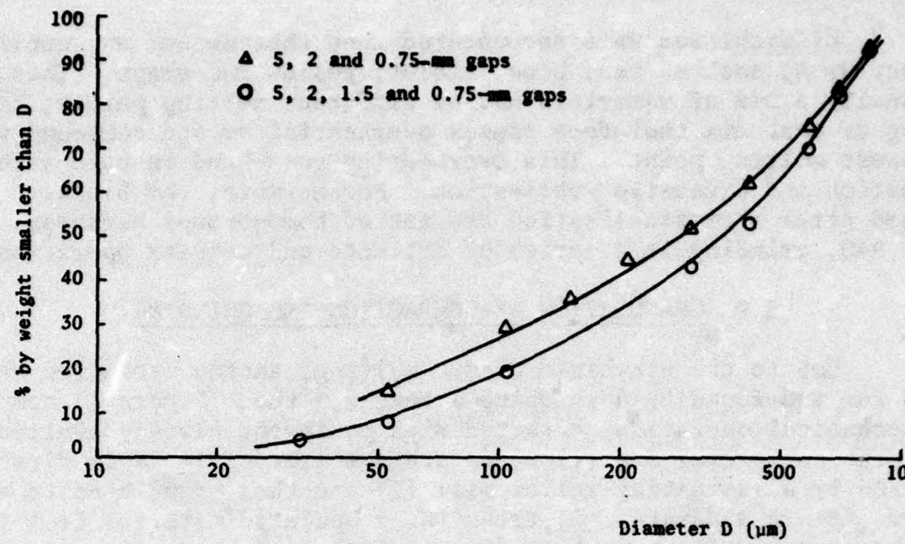


FIGURE 1 - Increase in fine particles when the number of passes between the rollers is reduced from 4 to 3 (Method I).

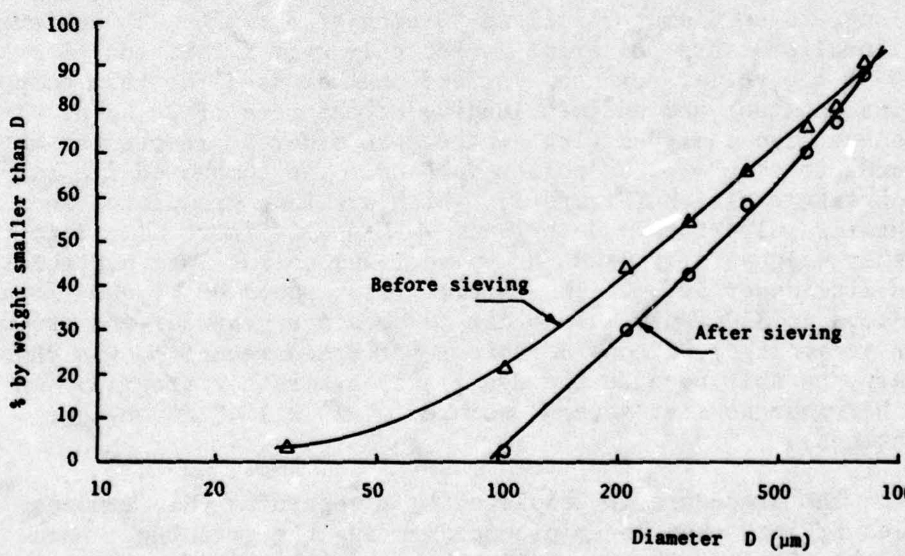


FIGURE 2 - Effect of sieving on the particle size distribution of 1-AAQ (Method I)

## 2.3 Constraints and disadvantages

Difficulties were encountered when that method was applied to other dyes, such as red, blue, violet, yellow and green. These dyes are usually a mix of materials having different melting points; the melting of that mix therefore causes overheating of the compound with the lowest melting point. This overheating goes hand in hand with some degradation and excessive sublimation. Furthermore, the blocks obtained after recrystallization are not of homogeneous hardness. Even with 1-AAQ, grinding is a series of delicate and complex operations.

# 3.0 GRANULATION BY COMPACTION AND GRINDING

Due to the drawbacks of dye melting, another solution was looked for and found by developing a second method (Figure 3) comprising only mechanical operations effected with equipment already available at DREV. The commercial dye (1) of an average size of 14 μm is first compacted by a laminating roller mill (2) and then granulated in a hammer crusher (4). A separator (5) frees the granulated material from its fine particles and directs them towards a cyclone (6) where they are recovered. The capacity of the installation is in the order of 20 kg/h.

## 3.1 Description of the equipment

The rolling machine used for compacting (Figure 4) has two 25-cm-long, 15-cm diameter rollers turning at 8 r/min. To achieve higher local pressure, material is fed only over a limited (15 cm) section of the roller surface. A feed chamber used for that purpose maintains constant and uniform loading at the rate of 20 kg/h. The rollers are also equipped with scrapers in order to remove the material that tends to stick to the roller surface. The compacted dye has the shape of little plates (Figure 3), which are then granulated in a Bantam Model hammer pulverizer made by Ducon-Mikropul (Figure 5). That type of crusher is generally used for pulverizing solids into particles of average size under 50 μm. The initial rotor speed of 14,000 r/min was reduced to 850 r/min. In order to obtain a granular end product (600 μm on average), a considerable rotor speed reduction was thus necessary, notably because the dye flakes are rather fragile. An HB-046 herringbone slot screen was used (1.17 x 12.8 mm oblique perforations).

The procedure is completed by a separator that removes particles of less than 75 μm produced during the grinding. When leaving the grinder, the dye falls through the separator shown in Figure 5, which consists of a chamber equipped with baffles designed to slow down the fall of the particles and to facilitate the elimination of fine particles. An air current carries the latter towards a 15-cm

cyclone where they are recovered and recirculated towards the compactor. Air circulation is provided by a (constant pressure) compressed-air jet through a venturi located in the hose linking the separator to the cyclone. This syphon creates a negative pressure in the separator and eliminates dust leakages. Furthermore, higher efficiency is achieved due to the larger airflow in the cyclone.

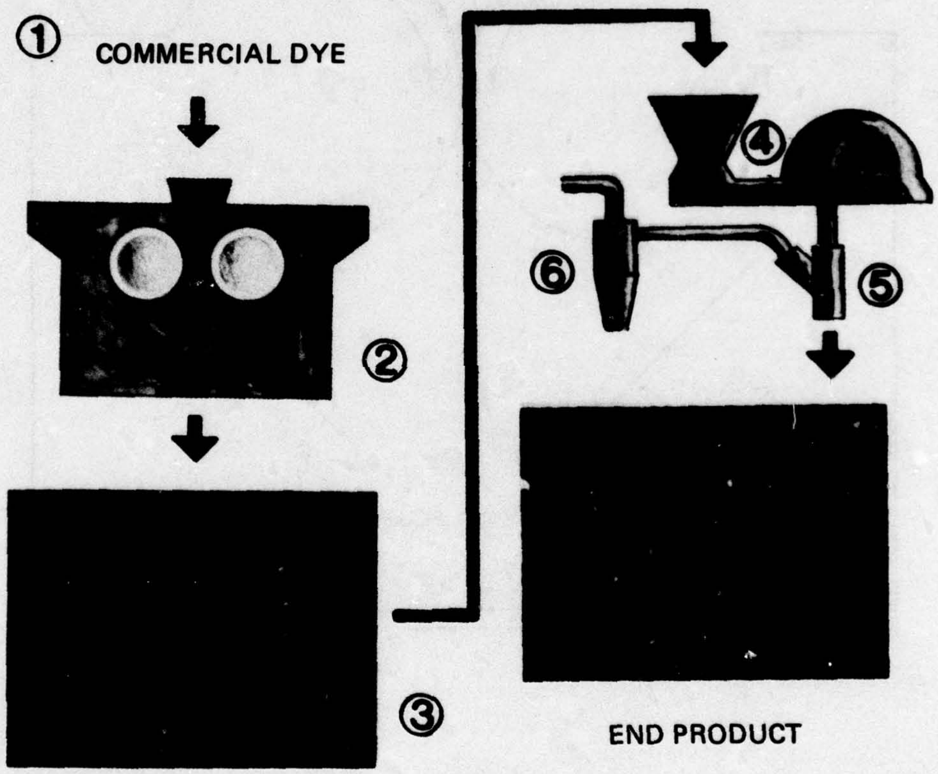


FIGURE 3 - Granulation by compaction and grinding

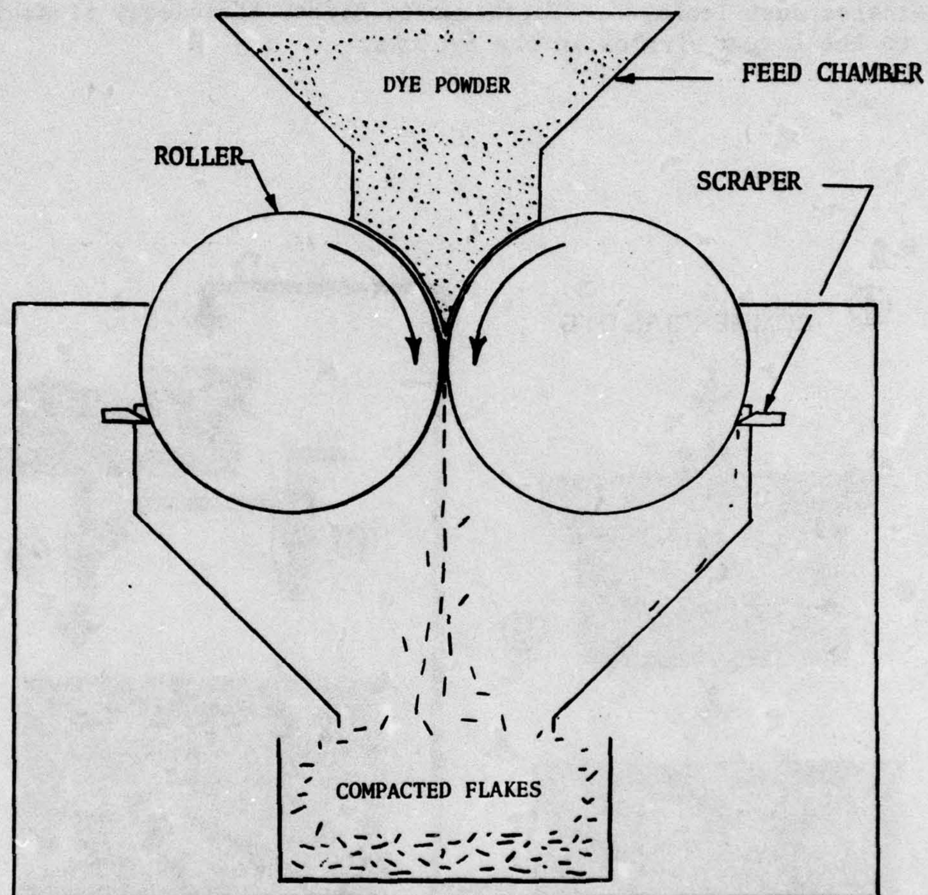


FIGURE 4 - Laminating roller mill

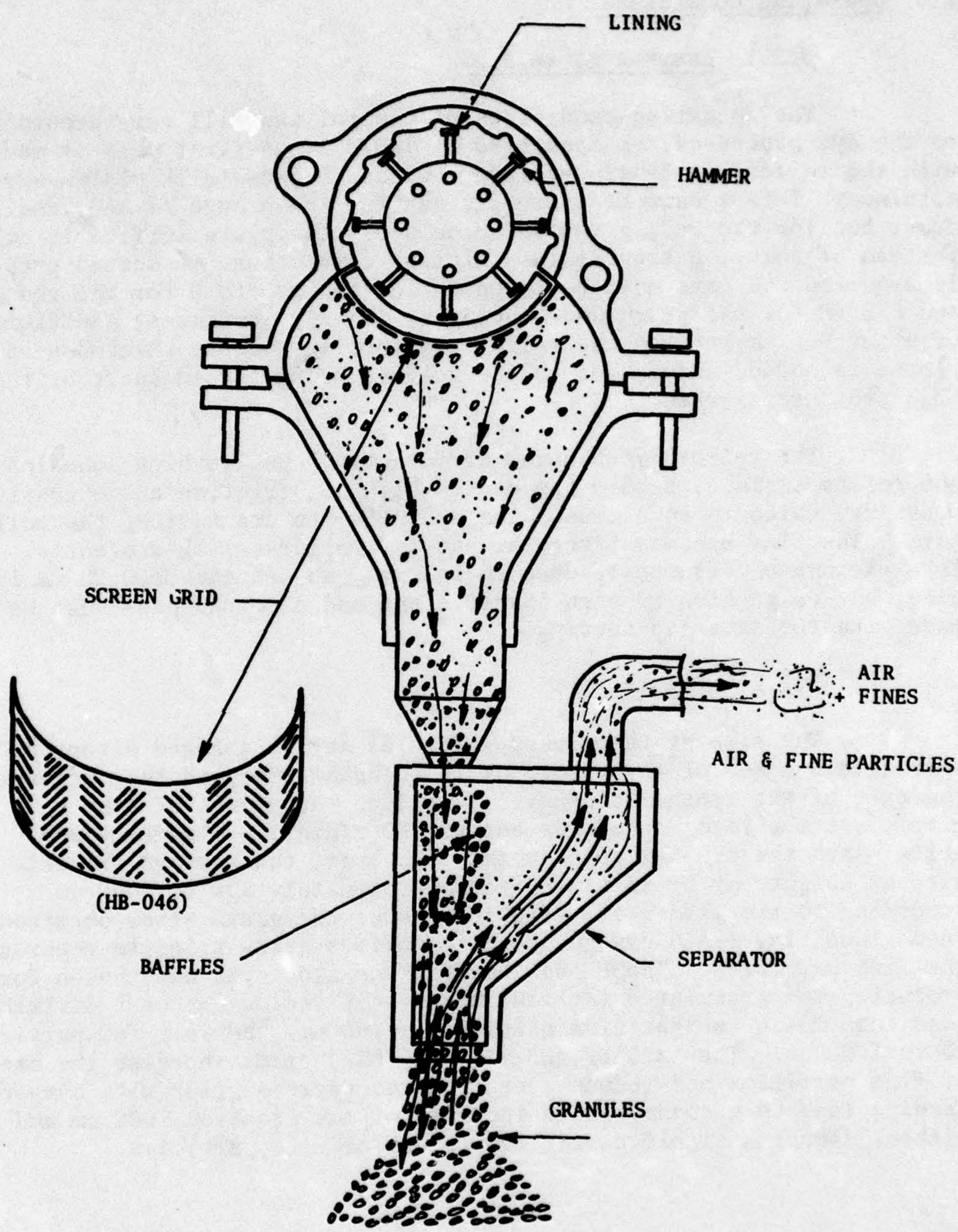


FIGURE 5 - Hammer crusher and separator

## 3.2 Operating conditions

### 3.2.1 Laminating roller

The operating conditions of the rolling mill vary according to the dye processed, as specified in Table I. A first pass is made with the rollers fully closed (gap = 0) and 0.6-mm-thick plates are obtained. This treatment is sufficient for the orange (1-AAQ) and violet dyes, but for the red, green and blue dyes, the plate surface is dull instead of shiny, a sign of insufficient compaction. A second processing is required and care must be taken to set the gap to 0 for the red dye and 0.6 mm for the green and blue dyes. In all instances, a difference of about 0.6 mm between the roller gap setting and the thickness of the plates is noted, and is due to the rollers being forced apart by the high pressure exerted.

The yellow dye behaves differently. Due to high adhesion to the roller surface, a zero gap causes high constriction and excessive pressure, which in turn causes the dye plate to crack along the roller axis. The flow becomes irregular and the rollers clash violently. This phenomenon disappears when an initial gap setting of 0.25 mm is used, but compaction is then insufficient and a second pass must be made with the same gap setting.

### 3.2.2 Grinder

The size of the ground particles may be changed either by varying the speed of the rotor, or by changing the grid that screens the exit of the crusher chamber. To reduce the amount of fine particles, the rotor speed was set at 850 r/min the minimum speed below which the crusher gets jammed. However, the average particle size by weight may be adjusted from approximately 500 to 1000 μm according to the grid used. Figure 6 shows the grain sizes obtained when processing 1-AAQ orange dye with various grids prior to separating the fine particles. The HB-046 herringbone slot grid was chosen for producing the granulated material because it yields the best distribution: less than 10% by weight fine particles (<100 μm) and very few particles above 1000 μm. The smaller (HB-035 and -027) grids increase the amount of fine particles and reduce average size, whereas grids with larger opening lead to a considerable increase of the fraction 1000 μm and up, without reducing significantly the amount of fine particles.

TABLE I

GAP BETWEEN THE LAMINATING ROLLERS

| Product | Pass 1 Gap (mm) | Pass 2 Gap (mm) | Final Flake Thickness (mm) |
|---|---|---|---|
| Orange (1-AAQ) | 0 | - | 0.63 |
| Orange (α -AAQ) | 0 | - | 0.56 |
| Red (1-MAAQ) | 0 | 0 | 0.58 |
| Violet | 0 | - | 0.63 |
| Yellow | 0.25 | 0.25 | 0.8 |
| Green | 0 | 0.6 | 1.2 |
| Blue | 0 | 0.6 | 1.2 |

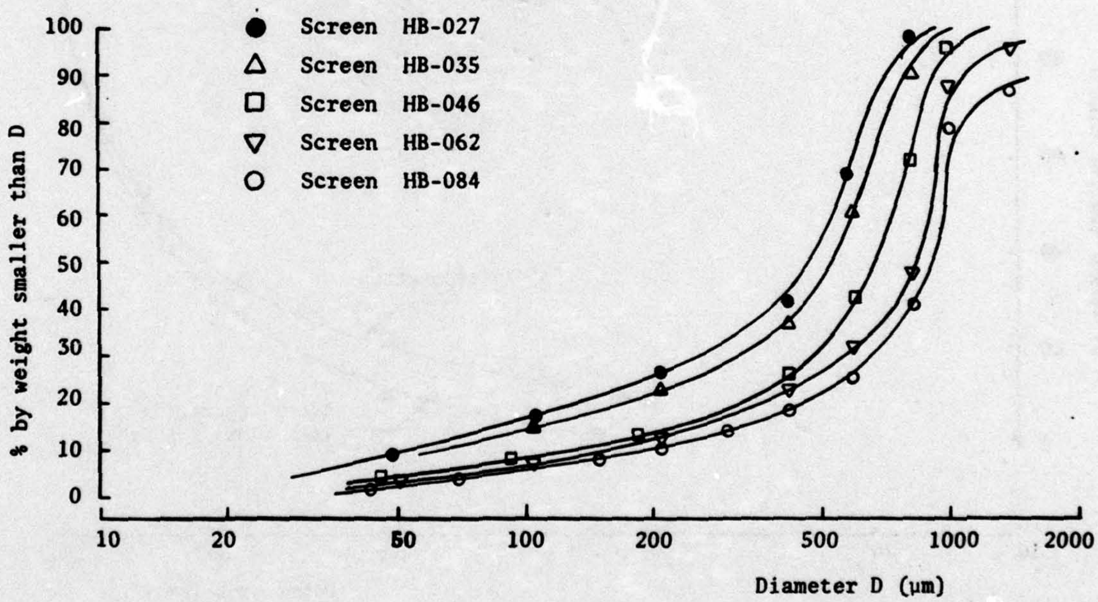


FIGURE 6 - Particle size distribution at the crusher exit before separation of the fine particles, for different screens

### 3.2.3 Separator

The separator is used to eliminate the fine particles produced by that method, as their presence in the end product is undesirable for two reasons: they are a source of dust during handling, and increase viscosity during the manufacture of composite mixes. This is due to the fact that the solids other than the dye, i.e. the $KClO_3$, the lactose and others saturate the suspension of fine particles because their size is less than 20 µm. All additional amounts of fine particles must thus be eliminated. The 8 to 16% by weight less than 100 µm at the crusher exit must be eliminated by means of a separator.

Figure 7 shows the effect of the separator in the case of the green dye, where a 3.5 $dm^3/s$ flow eliminated from the granulated material almost all particles less than 75 µm. Under the circumstances approximately 10% by weight of the particles was eliminated. A 3.0 $dm^3/s$ airflow is insufficient because it reduces efficiency considerably, and the end product contains 3 to 4% particles less than 50 µm. A 4 $dm^3/s$ flow removes almost all particles between 75 and 100 µm, but since their presence has no marked effect on mix viscosity, it is only used when processing the violet dye, which contains far more 100-µm particles than the others.

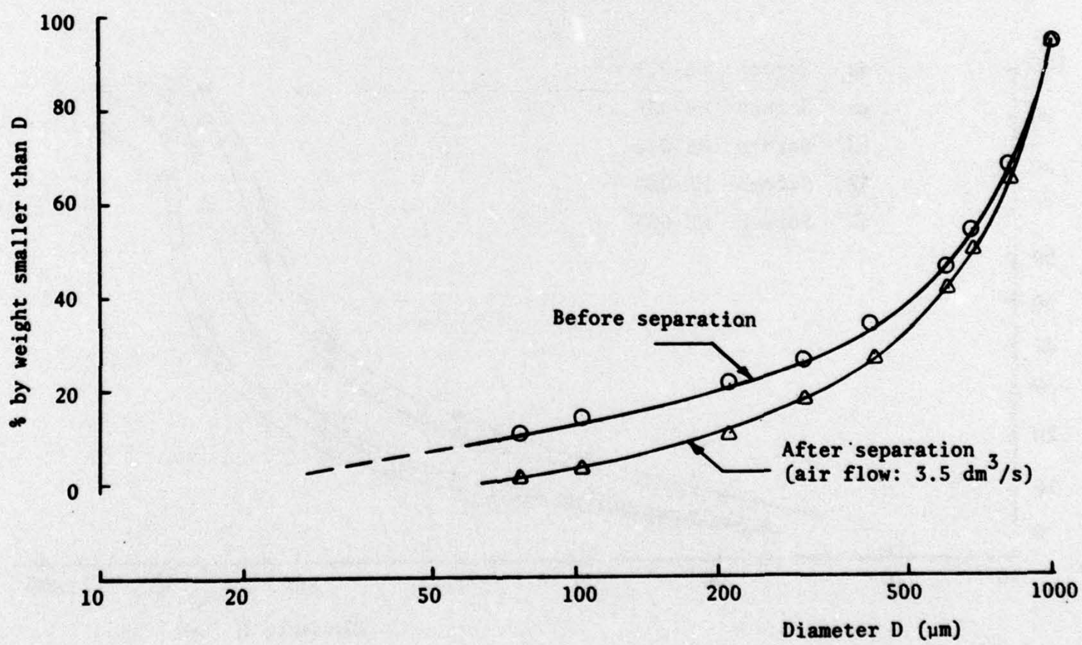


FIGURE 7 - Effect of the separator on particle size distribution

Figure 8 shows the variation of the distributions obtained for the various dyes, after fine particle separation. Airflow was always 3.5 $dm^3/s$, except for the violet dye, where 4.0 $dm^3/s$ was used. This method yields granulated material of average size, 500 to 750 µm, and completely free of fine particles 50 µm and less.

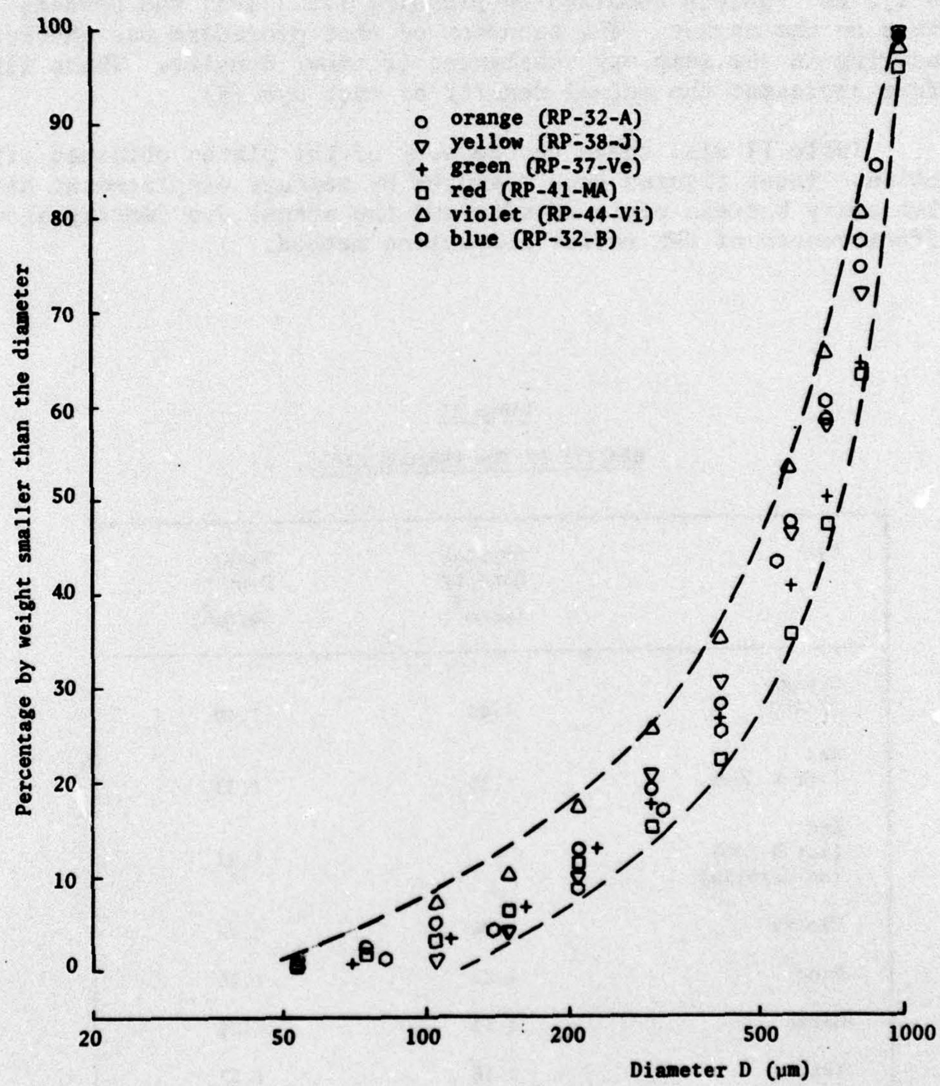


FIGURE 8 - End product particle size distributions

# 4.0 PROPERTIES AND ADVANTAGES OF THE GRANULATED MATERIAL

## 4.1 Dye Density

For the purpose of comparing and estimating the quality of the granulated dyes, density measurements were made on the various powders and granules. Dye density was determined (Table II) by measuring - by means of the mercury displacement method - the volume of the 1/2 $cm^3$ tablets obtained by pressing (55.1 mPa) the powders available on the market. The accuracy of that procedure was checked by measuring in the same way substances of known density. These figures therefore represent the actual density of each dye (4).

Table II also shows the density of the plates obtained after compaction. These figures were measured by mercury displacement as well. The similarity between plate density and the actual dye density shows the effectiveness of the roller compaction method.

TABLE II

DENSITY OF THE VARIOUS DYES

| Dye | Crystal Density ($g/cm^3$) | Flake Density ($g/cm^3$) |
|---|---|---|
| Orange (1-AAQ) | 1.43 | 1.40 |
| Red (lot R-300) | 1.33 | 1.33 |
| Red (lot R-302) (no dextrin) | - | 1.21 |
| Violet | 1.30 | 1.28 |
| Blue | 1.40 | 1.40 |
| Green | 1.33 | 1.29 |
| Yellow | 1.38 | 1.37 |

## 4.2 Apparent density and compaction index of the dyes

The apparent density of commercial powders and of granules was obtained by measuring - after 30 seconds' vibration - the volume taken up by a known weight of dye inserted into a 100 $cm^3$ graduated tube. The results are shown in Table III opposite the figures found in the literature (5,6). There is a considerable increase in apparent density when switching from fine material to type II granulated material. For instance, the respective figures are 0.45 and 0.83 $g/cm^3$ for the yellow dye. This Table also contains the corresponding compaction indices "ε", where "ε" is the ratio between apparent and real densities and represents the share of volume taken up by the solid. The results show that granulation has almost doubled the value of "ε".

Table IV gives the apparent densities and compaction indices achieved with the various 1-AAQ orange dye types. Thus "ε" is 0.66 for type II granulated material obtained by compaction and grinding, as opposed to 0.38 for 15 μm commercial dye. This is a 73% gain due to the large average size and to the broad range of particle sizes.

TABLE III

APPARENT DENSITY AND COMPACTION INDEX OF GRANULATED MATERIAL VS FINE POWDERS

| Dye | Apparent densities ($g/cm^3$) | | | Compaction index "ε" | |
|---|---|---|---|---|---|
| | Specified | Measured | | | |
| | Fine (10-20 μm) | Fine | Granulated Type II | Fine | Granulated Type II |
| Orange | - | 0.55 | 0.94 | 0.38 | 0.66 |
| Red | 0.30 ± 0.15 | 0.20 | 0.74 | 0.15 | 0.56 |
| Blue | 0.35 ± 0.15 | 0.20 | 0.72 | 0.31 | 0.56 |
| Green | 0.40 ± 0.20 | 0.48 | 0.75 | 0.44 | 0.55 |
| Yellow | 0.36 ± 0.10 | 0.45 | 0.83 | 0.33 | 0.60 |
| Violet | 0.35 ± 0.10 | 0.43 | 0.83 | 0.33 | 0.61 |

TABLE IV

COMPACTION INDEX "$\varepsilon$" FOR VARIOUS FORMS OF 1-AAQ

| Type | Avg size by weight (μm) | Apparent density ($g/cm^3$) | $\varepsilon$ |
|---|---|---|---|
| Commercial Powder | 15 | 0.55 | 0.38 |
| Granulated Mat. Method | 400 | 0.84 | 0.59 |
| Granulated Mat. Method II | 660 | 0.94 | 0.66 |

4.3 Effect of the granulated material on the compaction rate of smoke compositions

It was mentioned in the introduction that the viscosity of a liquid/solid suspension is affected by the compaction rate of the solid phase. In the present case, the solid phase contains 2 components, the dye and the fuel, which constitute the large and the small particles, respectively. Fuel means all solids other than the dye. The average size "d" of those solids is in the order of 15 μm. With the dye of average size "D" of 600 μm, the D/d ratio is 40. A ratio over 30 is generally considered to provide optimal compaction (2,7). The use of the granulated dye instead of the commercial dye in smoke compositions will thus lead to a significant drop in viscosity.

4.4 Effect of the granulated material on the smoke composition manufacturing characteristics

Figure 9 shows the end-of-mix viscosity variations for the various 1-AAQ grades. At 75% by weight solids, substitution of the fine-grained dye by type I (350 μm) granulated material reduced viscosity by a factor of 10. A mix containing 82% by weight solids could thus be obtained, even while staying below the 5 kP castability threshold. The type II (660 μm) granulated material was taken one step further, and mixes containing in excess of 85% by weight solids are now feasible.

Table V shows the end-of-mix viscosities recorded for the various dyes in mixes comprising 20% polybutadiene binder. The variations (1 kP for the orange dye and 4.5 kP for the red dye) can be ascribed to variations in compaction between dyes as well as to variations in dye concentration. The solid phase contains approximately 60% by volume granulated dye in all cases except for orange mixes (67% dye). Use of a dye other than orange thus means a departure from the optimum concentration of 65% by volume large particles, and an increase in mix viscosity due to the lower compaction rate of the solid phase.

## 5.0 CONCLUSION

The granulation of organic dyes used in smoke compositions has provided a solution to the problem of high mix viscosity. It is now feasible to prepare smoke compositions containing up to 85% by weight solids and having an end-of-mix viscosity of less than 5 kilopoises.

A first method of granulation by fusion, crystallization and grinding gave good results with the orange dye (1-AAQ), but fusion created difficulties with the other dyes because they are mixes.

The second method, basically mechanical, involved compaction and grinding, and was used successfully with all the dyes tried, i.e. yellow, orange, red, violet, blue and green. It can be used in a continuous process to produce granulated material virtually devoid of porosity and of a size adjustable at will. Whereas the maximum solids loading of orange compositions was in the order of 75% by weight (10-12 kP) with the powder dye available on the market, it is now possible to obtain mixes containing up to 85% by weight solids and having an end-of-mix viscosity less than 3.5 kP, with granulated material of an average size of 650 μm.

Equivalent improvements were obtained with the other dyes, but their end-of-mix viscosity remains higher than that of 1-AAQ. However, at virtually identical grain size, the castability thresholds are all above 80% by weight.

Used in the same proportions as the dye available on the market, the granulated material did not affect combustion or smoke quality (color and volume). However, the improved smoke composition manufacturing properties made it possible to raise the smoke composition solids loading by 10%, thus providing a considerable improvement in overall smoke generator efficiency.

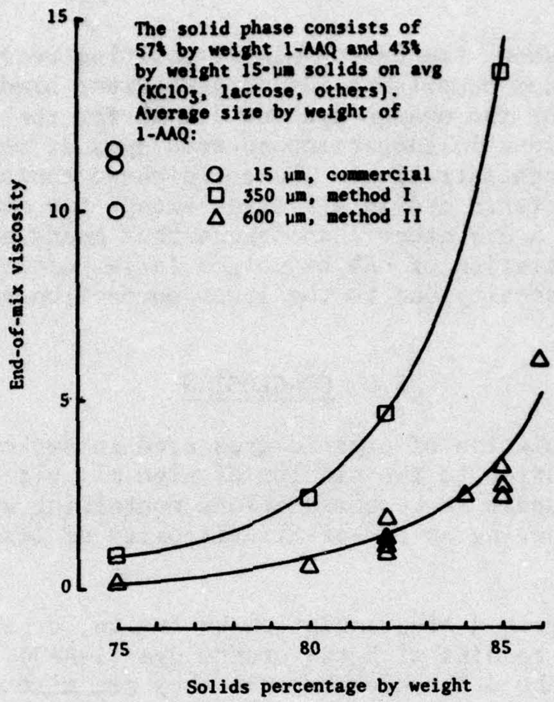


FIGURE 9 - Mix viscosity variation according to the size of 1-AAQ dye

TABLE V

END-OF-MIX VISCOSITIES (kP) FOR DIFFERENT DYES

| Dye | Viscosity (end-of-mix)* |
|---|---|
| Orange (1-AAQ) | 0.5 @ 1.0 |
| Red (1-MAAQ) | 4.5 |
| Violet | 2.7 |
| Yellow | 2.3 |
| Green | 3.9 |
| Blue | 2.5 |

* Except for blue dye, these viscosities are an average for 10 mixes containing 20% by weight binder. Type II dye is 50% of the solid phase. Above figures at a temperature of 60°C.

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## APPENDIX

### Description of the dyes (5,6)

ORANGE: 1-amino-anthraquinone, (1-AAQ), $C_{14}H_9NO_2$
C.I. 37275

RED: 1-methyl-amino-anthraquinone, (1-MAAQ), $C_{15}H_{11}NO_2$

C.I. 60505, C.I. Disperse Red 9
(Lot R-300, with dextrin)
(Lot R-302, without dextrin)

BLUE: 1,4-di(methylamino) anthraquinone, $C_{16}H_{14}N_2O_2$

C.I. 61500, C.I. Disperse Blue 14

GREEN: Dye Mix, Smoke Green 4.
Mix of: - Solvent Green 3 C.I. 65656
- Vat Yellow 4 C.I. 59100
- Benzanthrone -

YELLOW: Dye Mix, Smoke Yellow 6
Mix of: - Vat Yellow 4 C.I. 59100
- Benzanthrone

VIOLET: Dye Mix, Violet
Mix of: - Disperse Red 9 C.I. 60505
- 1,4-diamino-2,3-dihydroanthraquinone

(C.I.: Color Index).

CRDV R-4053/78 (A) (NON CLASSIFIE)

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"Granulation de teintures organiques pour compositions fumigènes coulables"
par G. Couture et A. Roy

La préparation de compositions fumigènes coulables, ayant une viscosité de fin de mélange de moins de 5 kilopoises (kP) pour des concentrations en solides de plus de 80% en poids, ne peut se faire que si on utilise des colorants granulés. Comme les teintures organiques entrant dans ces compositions ne se trouvent sur le marché que sous forme de poudres pulvérulentes, on a mis au point deux procédés de granulation. Le premier, par voie de fusion/cristallisation/broyage, est efficace pour les colorants ne contenant qu'un seul composé, comme le colorant orange qui a un point de fusion unique. Par contre le second procédé, par compactage/broyage, a donné de bons résultats avec tous les colorants étudiés; on peut l'utiliser en continu et produire économiquement du granulé de la grosseur désirée. On met également en lumière les propriétés du granulé et ses avantages au point de vue de la mise en oeuvre des mélanges fumigènes. (NC)

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La préparation de compositions fumigènes coulables, ayant une viscosité de fin de mélange de moins de 5 kilopoises (kP) pour des concentrations en solides de plus de 80% en poids, ne peut se faire que si on utilise des colorants granulés. Comme les teintures organiques entrant dans ces compositions ne se trouvent sur le marché que sous forme de poudres pulvérulentes, on a mis au point deux procédés de granulation. Le premier, par voie de fusion/cristallisation/broyage, est efficace pour les colorants ne contenant qu'un seul composé, comme le colorant orange qui a un point de fusion unique. Par contre le second procédé, par compactage/broyage, a donné de bons résultats avec tous les colorants étudiés; on peut l'utiliser en continu et produire économiquement du granulé de la grosseur désirée. On met également en lumière les propriétés du granulé et ses avantages au point de vue de la mise en oeuvre des mélanges fumigènes. (NC)

CRDV R-4053/78 (A) (NON CLASSIFIE)

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DREV R-4053/78 (E) (UNCLASSIFIED)

Research and Development Branch, DND, Canada.
DREV, P.O. Box 880, Courcelette, Que. GOA 1RO

"Granulation of organic dyes for castable smoke compositions"
by G. Couture and A. Roy

Processing of castable smoke compositions having an end-of-mix viscosity less than 5 kilopoises and a solids loading greater than 80% by weight is only possible when granulated dyes are used. Since the organic dyes used in these compositions are commercially available only as very fine powders, two methods of granulation have been developed. The first method, by fusion, crystallization and grinding, is useful for single component dyes, such as orange, where there is a single melting point. The second method, by compaction and grinding, has, on the other hand, given good results with all the dyes tried and can be used in a continuous process to economically produce granulated material of the desired size. Also discussed are the properties of the material and its advantages with respect to the manufacture of smoke compositions. (U)

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